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MICROSTRUCTURAL ANALYSIS OF 316LN AUSTENITIC STAINLESS STEEL BY SALT BATH NITRIDING PROCESS

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ABSTRACT

Austenitic stainless steel 316LN was subjected to salt bath nitriding at a temperature of 565°C for a period of 60, 80, 100 and 120 minutes. Influence of case depth based on nitriding time was investigated in the present study. Microstructural studies revealed an additional layer was developed on the surface with case depth varying from 7.1µm to 15.9µm. X-ray diffraction results confirm the formation of iron nitrides due to nitriding process. Growth of the nitrided layer is mainly because of nitrogen diffusion into the matrix in accordance with the parabolic rate law. Final study reveals that case depth of nitriding has a saturation limit and further increase in nitriding time doesn't yield any change in case depth. Vickers hardness measurements were used to study the influence of nitriding with varying the time.

KEYWORDS: Austenitic Stainless Steel, 316LN, Salt Bath Nitriding & Case Depth

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Original Article

INTRODUCTION

Austenitic stainless steel (ASS) are used in a variety of outdoor application like chemical, coal, oil industries etc. due to its excellent corrosion resistance, ductile behavior and nonmagnetic properties [1-3]. Major disadvantage of ASS is its low hardness, fatigue resistance and poor tribological properties [4, 5]. To enhance the hardness and tribological behavior of ASS so that it can be used in a wide range of application chemical surface treatment such as nitriding, carburizing and nitrocarburizing are carried out [6, 7]. To harden steels and modify the surface where in improving its tirbological properties salt bath nitriding is used as one of the technique. Achievement of good wear resistance, high fatigue resistance and corrosion resistance can be accomplished by this environment friendly technique. Salt bath treatment has the advantage of low operating cost, energy efficient and stability of the layer deposited on the surface [8]. This process can be carried out at a temperature level of 580-650°C. Recent advancement in salt bath nitriding is the development of low temperature process. This low temperature process enhances the enhancement of wear resistance without compensating its hardness. In addition to this low temperature nitriding reduces distortion of materials.

Nitriding is done mainly to increase the wear resistance, enhancing the fatigue properties and the corrosion resistance of the material. Nitriding is done mainly to diffuse nitrogen into the material. Nitriding is a best substitute for chromium plating, phosphorescing etc.. Potassium nitrate (KNO_3) bath is used to diffuse nitrogen into the material [9]. The dissolution of KNO_3 is according to the following reaction.

$$2KNO_3 \rightarrow K_2O + 2NO_2 + \frac{1}{2}O_2 \quad (1)$$

$$2KNO_3 \rightarrow K_2O + \frac{5}{2}O_2 + N_2 \quad (2)$$

From the above equations it can be seen that decomposition of KNO_3 on heating leads to the liberation of nascent nitrogen which will transform to molecular nitrogen (N_2) [10]. This nascent nitrogen will diffuse into the steel specimens. Nitrogen solubility in iron is greater compared to that of carbon at a temperature ranging between 20°C and 600°C, thus reducing the tendency of precipitate formation. Nitrogen forms interstitial solid solution strengthening with iron and its alloys which is superior to that of carbon. Moreover nitrogen increases corrosion resistance, creep strength and wear resistance to iron and its alloys. ASS is a known material which is difficult to nitride. Lack of knowledge persists around nitriding of steels on metallurgical aspects at low temperatures.

The aim of this current study is to examine the processing time of salt bath nitriding by validating the case depth using micro structural, X-ray diffraction (XRD) and energy dispersive spectroscopy analysis.

EXPERIMENTAL

Plates of 316 LN grade ASS were cut using electro discharge machining into small pieces of length 30mm, breadth 20mm and thickness of 5mm. Elemental composition of 316 LN materials is shown in table 1. Before placing inside the nitriding furnace all the specimens were subjected to de-greasing and cleaning the surface with trichloro ethylene. The 316 LN ASS samples for nitriding were dipped in molten salt bath of KNO_3 at 565°C for a period of 60, 80, 100 and 120 minutes and then cooled in air to room temperature, where the name of the samples are SBN1, SBN2, SBN3 and SBN4 respectively. After nitriding samples were chemically treated and ultrasonically cleaned in alcohol for 15 min. The nacent nitrogen utilized for nitirding comes from the dissociation of KNO_3 as shown from equation 2. The nitriding parameters were given in table 2. Post nitriding structural changes of the nitrided layer of ASS samples were investigated using cross-sections of the nitrided ASS for optical microscopy. X-ray diffraction (XRD) analysis was used to determine the different phases present after nitirding process with CuK alpha radiation. To measure the hardness value Vickers hardness were used to evaluate for all the samples with a applied load of 10 g/f for a dwell time of 15 sec by using diamond intender.

Table 1: Chemical Composition for 316LN

	Cr	Ni	Mn	Mo	C	S	Si	P	Fe
316 LN	18	14	2	3	0.03	0.1	0.75	0.25	Rest

Table 2: Salt Bath Nitriding Parameters for 316 LN ASS

Nitriding temperature	565°C
Nitriding time for four samples	60, 80, 100 and 120 minutes
Salt bath used	Potassium nitrate

RESULTS AND DISCUSSIONS

Microstructural Analysis

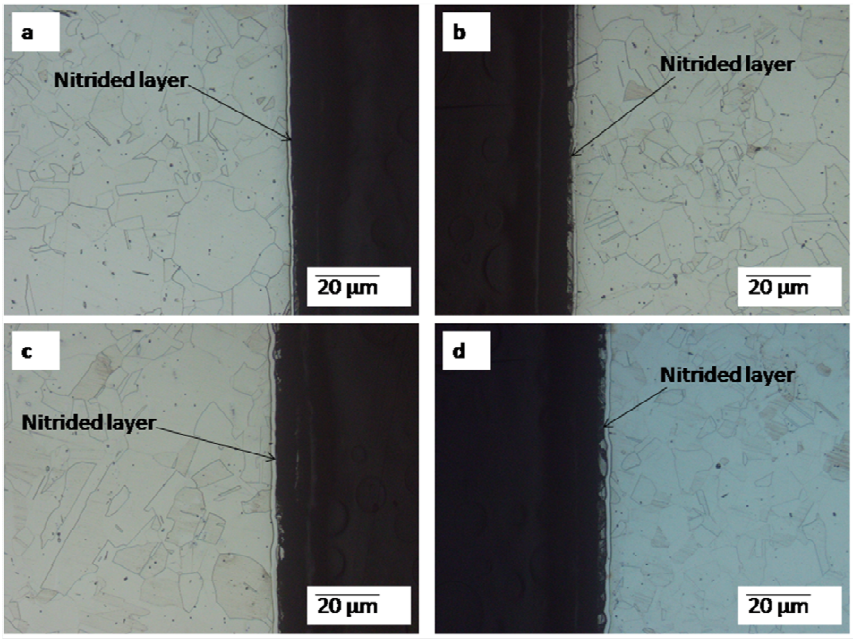

Figure 1: Optical Micrograph of Salt Bath Nitirided 316 LN ASS with Nitriding Time a) 60 min, b) 80 min, c) 100 min and d) 120 min

It is evident from figure 1 which reveals the microstructure of 316 LN material. Cross sectional microstructure of the 316 LN nitrided samples shows the nitrided layer which is marked and separated by a line. It can be further seen from the microstructure the case depths of the nitrided layer and case depths were measured using micron marker whose values are given in figure 2. These layer are corrosion resistant and the microstructure is revealed by very harsh etchant Marble's reagent. It can be seen from figure 2 that the nitrided case depth increases with increase in nitriding time. But it can be seen that once the saturation level is reached there will not be any increase in nitirding case depths. SBN4 sample is having similar case depth of SBN3 sample, which shows that for 316 LN ASS the maximum limitation of case depth is 15.9 µm. Further increase in nitriding will not have any significant increase in case depth of the nitrided layer.

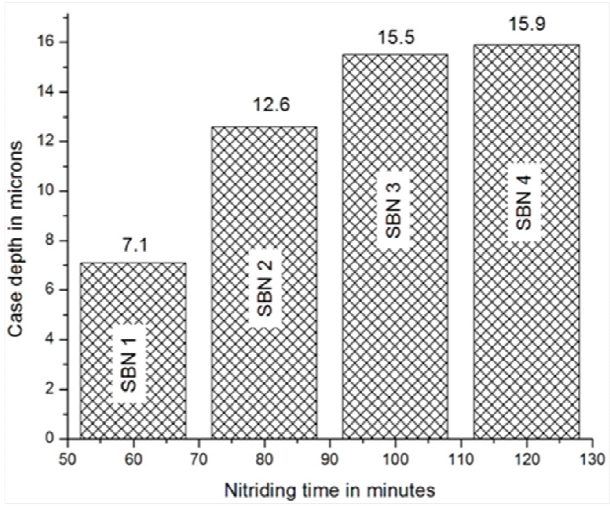

Figure 2: Case Depth of the Nitrided Samples

X-Ray Diffraction Analysis

Figure 3 & 4 shows the diffraction pattern of the nitrided and untreated 316 LN ASS samples. In order to show the difference between the nitrided and non-treated sample the conventional XRD of 316 LN ASS is shown for comparison. From the XRD pattern it can be seen that the phase composition of the nitrided layer on 316 LN depends on nitriding time [11,12,13]. From figure 4 it can be seen that the untreated 316 LN ASS is dominated by austenitic phase which is of FCC structure. After salt bath nitirding of 316 LN ASS the microstructure has peaks of iron nitrides. As nitrogen is an austenitic stabilizer and it forms rich nitrogen layer which tends to form a FCC structure rather than BCC structure. When the diffusion of nascent nitrogen takes place FCC crystal structure is helpful in the transformation of remaining alpha to austenitic which is namely the "S" phase. From the XRD results from figure 3 & 4 nitriding at 565°C shows a peak values at diffraction angle 2θ of 42° and 49° respectively. As the nitriding time increases, more number of nitride peaks increases which is evident from the figure 3.

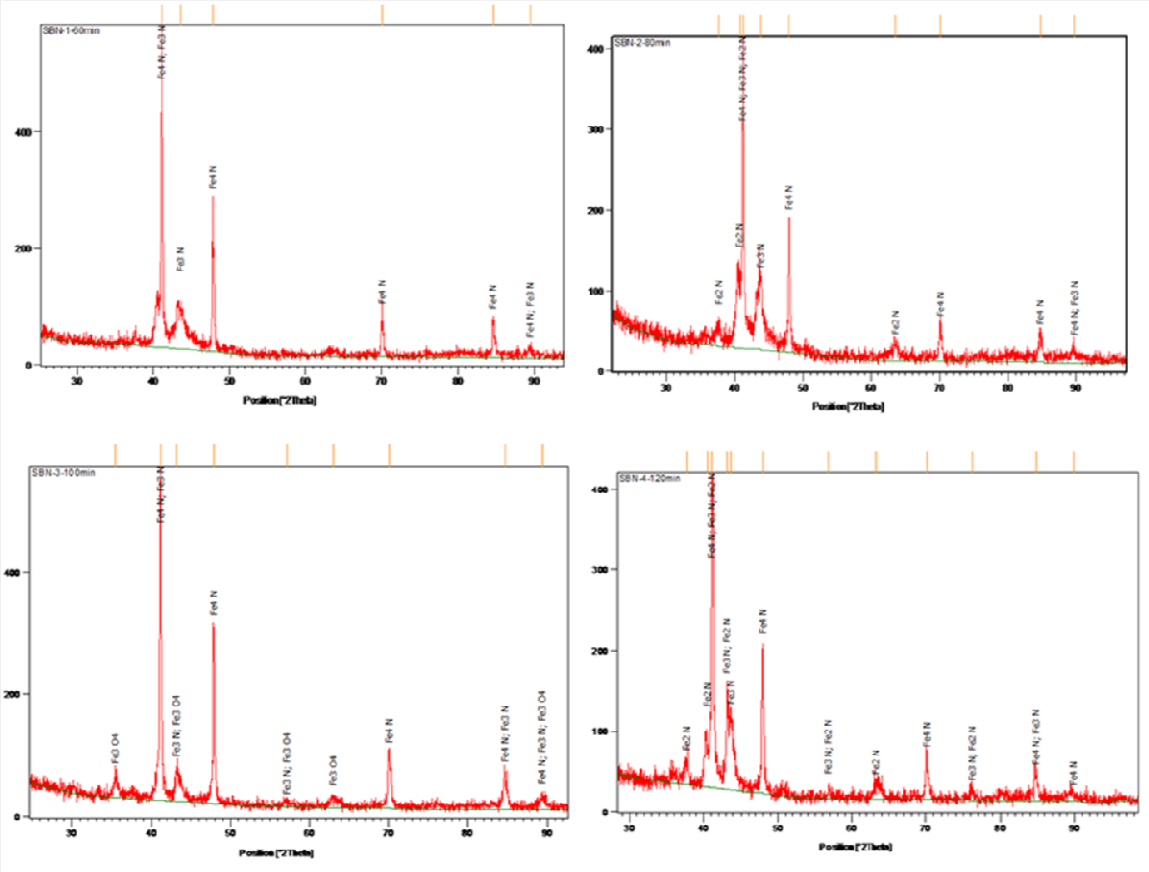

Figure 3: X-ray Diffraction of the Salt Bath Nitrided Samples

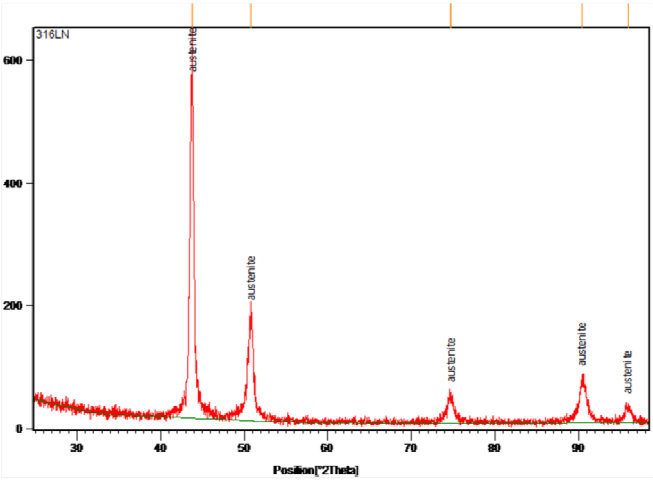

Figure 4: X-ray Diffraction of 316 LN ASS

Hardness Measurement

Hardness measurement was calculated for both the untreated and nitrided 316 LN ASS by using Vickers hardness measurement. It is found that 316 LN ASS is having a hardness of 230$Hv_{0.01}$. The maximum hardness value of the specimens SBN1, SBN2, SBN3 and SBN4 were found to be 1280 $Hv_{0.01}$, 1290 $Hv_{0.01}$, 1400 $Hv_{0.01}$ and 1410 $Hv_{0.01}$ respectively. It can be further noted that hardness value decreases as the distance from the surface increases towards the core. This shows that the diffusion of nitrogen concentration decreases as we move towards the core.

CONCLUSIONS

In the present study salt bath nitriding of 316 LN ASS has the following inferences

- It can be seen that as the time for nitriding increases the case depth increases. Maximum case depth of 15.9 µm is revealed after nitriding of 120 hrs.
- From the microstructure it can be seen that there is a saturation limit for nitriding time. If the saturation limit is attained there will be no significant increase in case depth which is evident from nitriding of SBN 3 & SBN 4 samples. This is also evident from the hardness values as there is not much change between SBN 3 & SBN 4 samples.
- XRD analysis reveals the presence of austenitic phase in the samples, moreover from the XRD results it shows the presence of iron nitrides increases as the nitriding time increases.
- Hardness value of salt bath nitrided specimen (SBN 4) has improved by about 84% when compared with untreated specimen.

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